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Boek et al.

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(54) **METHODS OF FORMING HIGH-DENSITY ARRAYS OF HOLES IN GLASS**

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See application file for complete search history.

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(57) **ABSTRACT**

A method of fabricating a high-density array of holes in glass is provided, comprising providing a glass piece having a front surface, then irradiating the front surface of the glass piece with a UV laser beam focused to a focal point within ± 100 μm of the front surface of the glass piece most desirably within ± 50 μm of the front surface. The lens focusing the laser has a numerical aperture desirably in the range of from 0.1 to 0.4, more desirably in the range of from 0.1 to 0.15 for glass thickness between 0.3 mm and 0.63 mm, even more desirably in the range of from 0.12 to 0.13, so as to produce open holes extending into the glass piece **100** from the front surface **102** of the glass piece, the holes having an diameter the in range of from 5 to 15 μm , and an aspect ratio of at least 20:1. For thinner glass, in the range of from 0.1-0.3 mm, the numerical aperture is desirably from 0.25 to 0.4, more desirably from 0.25 to 0.3, and the beam is preferably focused to within ± 30 μm of the front surface of the glass. The laser is desirable operated at a repetition rate of about 15 kHz or below. An array of holes thus produced may then be enlarged by etching. The front surface may be polished prior to etching, if desired.

20 Claims, 5 Drawing Sheets

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PCT Pub. Date: **Jun. 7, 2012**

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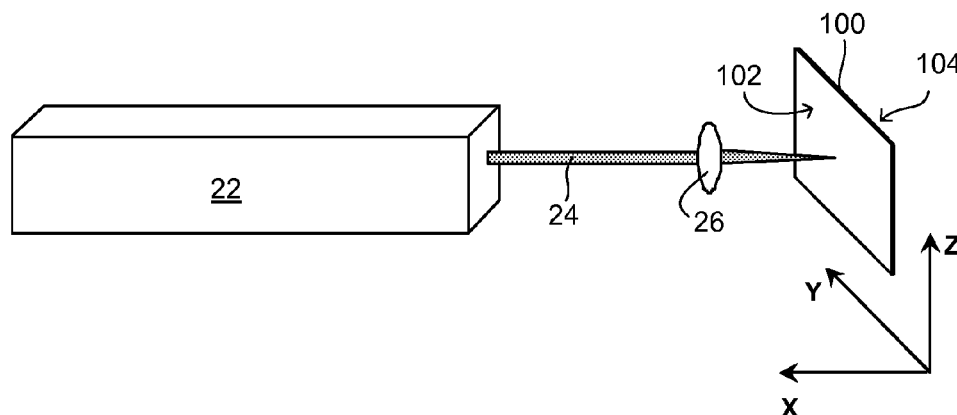
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(2013.01); **C03C 15/00** (2013.01); **G02B**
6/1225 (2013.01); **G02B 6/136** (2013.01)
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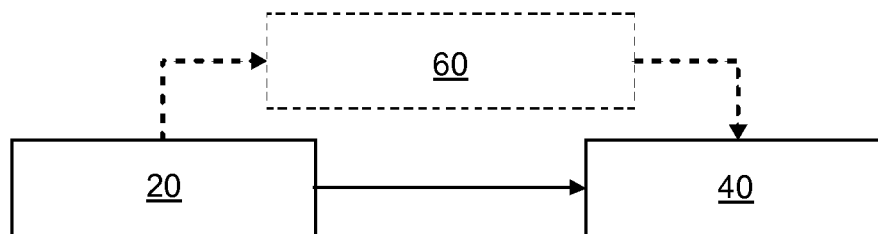


Figure 1

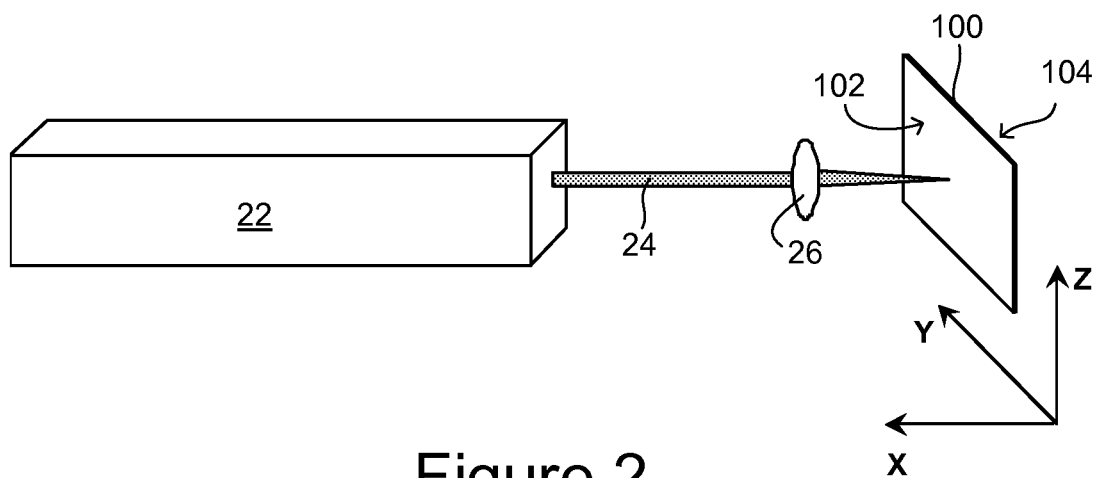


Figure 2

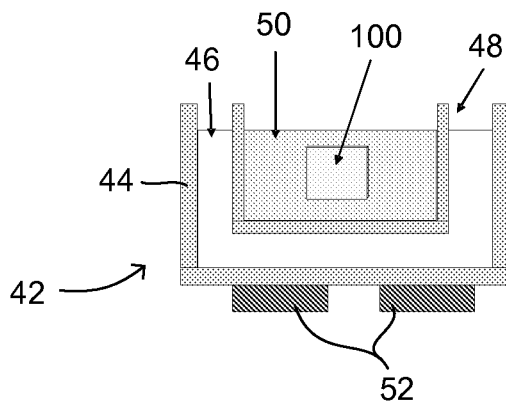


Figure 3

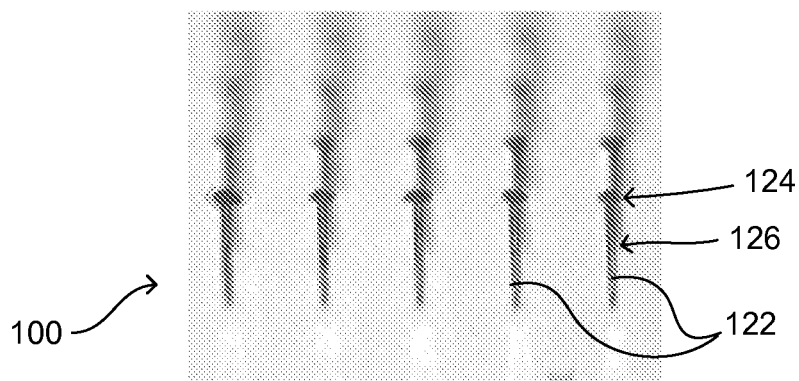


Figure 4

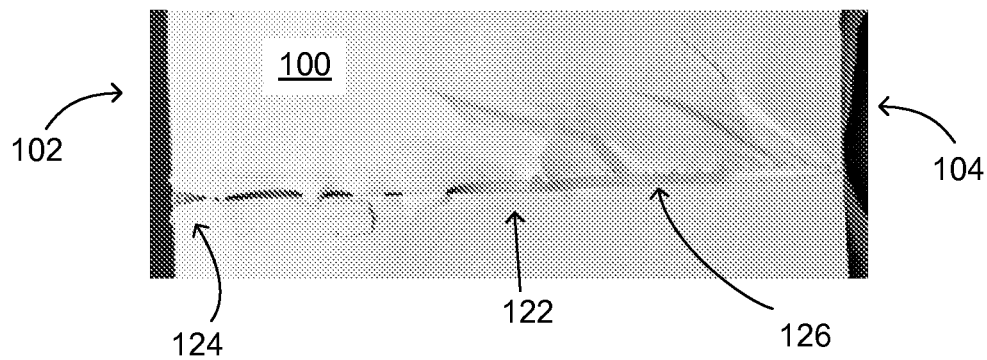


Figure 5

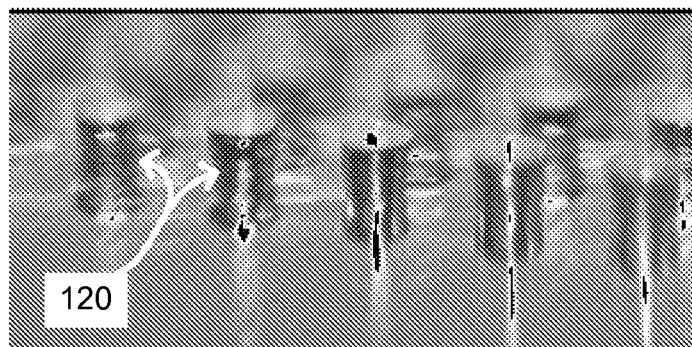


Figure 6

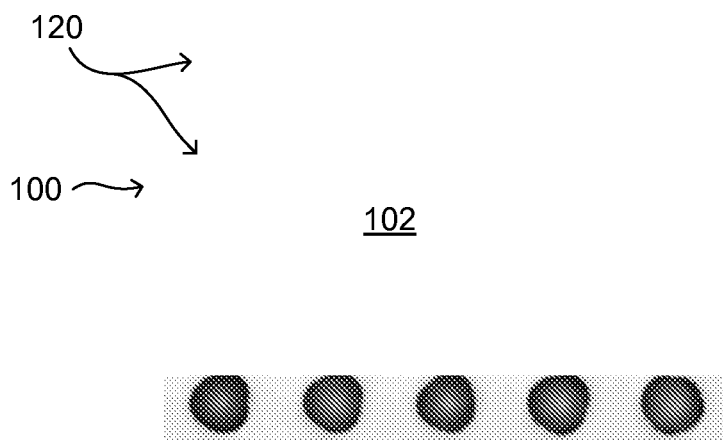


Figure 7

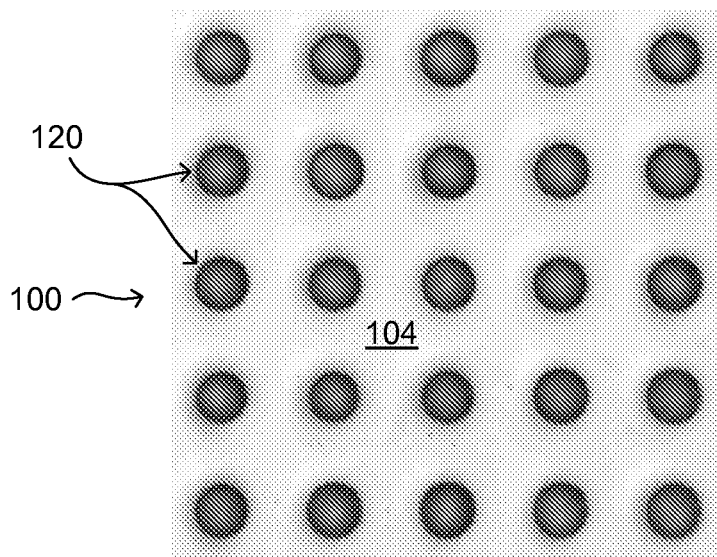


Figure 8

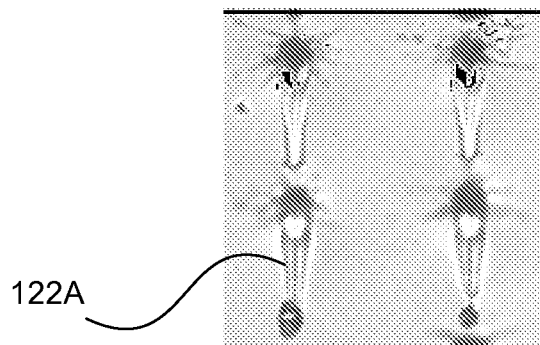


Figure 9

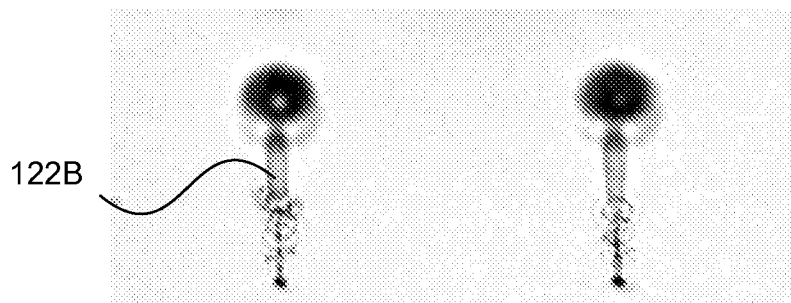


Figure 10

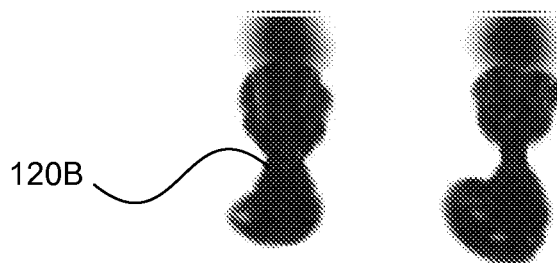


Figure 11

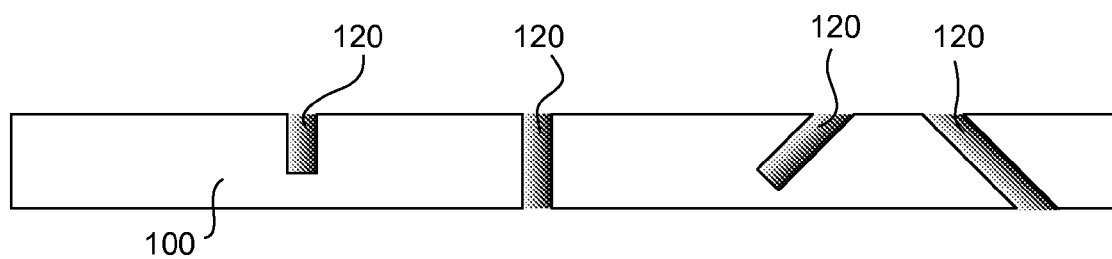


Figure 12

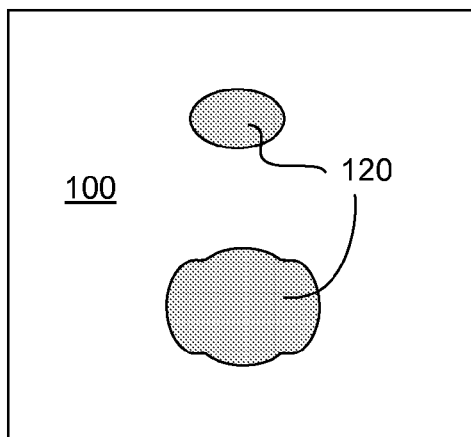


Figure 13

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METHODS OF FORMING HIGH-DENSITY ARRAYS OF HOLES IN GLASS

PRIORITY

The present application claims priority to U.S. application Ser. No. 61/418,152 filed Nov. 30, 2010.

FIELD

The present disclosure relates to methods of fabricating high-density arrays of holes in glass, particularly high-density arrays of through-holes, and also particularly high-density arrays of high aspect ratio holes.

BACKGROUND AND SUMMARY

A previously demonstrated process for making such dense arrays of holes in glass is disclosed in U.S. Pat. No. 6,754,429. The disclosed method involves glass exposure with a femto-second laser followed by acid etching. It relies on selective etching in which the laser-damaged glass etches at a significantly higher rate than the rest of the glass. Although technically sound, this approach utilizes expensive and maintenance-hungry femtosecond laser technology, and the laser exposure process is relatively slow.

According to US Patent Publication No. 20030150839, tapered (conical) holes 120-130 μm in diameter may be made by laser ablation followed by acid etching to remove surface defects and chips. The disclosed process requires an ion-exchange step before laser irradiation. Irradiation conditions beyond laser spot size and fluence are not disclosed.

US Patent Publication 20090013724 describes hole formation by laser irradiation and acid etching in glasses of various compositions. Lasers with wavelengths 355 nm and 266 nm were used. The recommended (numerical) beam aperture is $\text{NA} < 0.07$ and the focus is disclosed as either within the glass or behind the back surface. Hole profile and placement accuracy are not specifically addressed.

What is needed is a relatively low-cost and reliable process for forming relatively small holes at relatively tight minimum pitch, with good positioning accuracy and reasonably small variation in diameter throughout the depth.

According to one aspect of the present disclosure, fabricating a high-density array of holes in glass, the method comprises providing a glass piece having a front surface, then irradiating the front surface of the glass piece with a UV laser beam focused to a focal point within $\pm 100 \mu\text{m}$ of the front surface of the glass piece most desirably within $\pm 50 \mu\text{m}$ of the front surface. The lens focusing the laser has a numerical aperture desirably in the range of from 0.1 to 0.4, more desirably in the range of from 0.1 to 0.15 for glass thickness between 0.3 mm and 0.63 mm, even more desirably in the range of from 0.12 to 0.13, so as to produce open holes extending into the glass piece **100** from the front surface **102** of the glass piece, the holes having an diameter the in range of from 5 to 15 μm , and an aspect ratio of at least 20:1. For thinner glass, in the range of from 0.1-0.3 mm, the numerical aperture is desirably from 0.25 to 0.4, more desirably from 0.25 to 0.3, and the beam is preferably focused to within $\pm 30 \mu\text{m}$ of the front surface of the glass. The laser is desirable operated at a repetition rate of about 15 kHz or below, and generally of sufficient irradiation duration to form an open hole extending just up to a back surface of the glass piece. An array of holes thus produced may then be enlarged by etching. The front surface may be polished prior to etching, if desired.

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Variations of the methods of the present disclosure are described in the text below and with reference to the figures, described in brief immediately below.

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BRIEF DESCRIPTION OF THE DRAWINGS

The following detailed description of specific embodiments of the present disclosure can be best understood when read in conjunction with the following drawings, where like structure is indicated with like reference numerals and in which:

FIG. **1** is a flow diagram representing basic steps of a presently preferred method of the present disclosure;

FIG. **2** is a diagrammatic perspective view of a laser irradiation apparatus useful in the methods disclosed herein;

FIG. **3** is a diagrammatic cross section of an etching apparatus useful in the methods disclosed herein;

FIG. **4** is an image of holes in a glass piece produced according to methods of the present disclosure;

FIG. **5** is an image of fractured glass piece fractured so as to show a partial cross-section of a hole produced according to methods of the present disclosure;

FIG. **6** is an image of holes in a glass piece produced according to methods of the present disclosure, after an etching step;

FIG. **7** is a plan-view image of holes in a glass piece produced according to methods of the present disclosure viewed from a front surface of the glass piece;

FIG. **8** is a plan-view image of holes in a glass piece produced according to methods of the present disclosure viewed from a back surface of the glass piece;

FIG. **9** is an image of holes in a glass piece produced according to a comparative methods;

FIG. **10** is an image of holes in a glass piece produced according to a comparative methods;

FIG. **11** is an image of holes in a glass piece produced according to comparative methods, after an etching step;

FIG. **12** is a schematic cross-section of a glass sheet **100** showing alternative types of holes **120**; and

FIG. **13** is a schematic plan view of a glass sheet **100** showing alternative types of hole shapes of holes **120**.

DETAILED DESCRIPTION

According to one aspect of the present disclosure, holes of 200 μm or less diameter on a minimum pitch of not more than 300 μm , with variation in diameter limited to 10 μm or less, desirably 5 μm or less, and with placement (hole center) positional variation limited to 8 μm or less, desirably 4 μm or less, are formed in a thin sheet of glass, desirably less than 0.8 mm thick, preferably in the range of 0.1 to 0.63 mm thick. The thinnest or "waist" diameter of the holes is not less than 65% of the diameter of the opening at the surface, desirably not less than 80%.

These results and other beneficial results can be obtained by the methods of the present disclosure, which will be described with general reference to FIGS. **1-3**. According to one embodiment of the method of fabricating a high-density array of holes in glass, the method comprises providing a glass piece **100** having a front surface **102**, then irradiating the front surface of the glass piece **100** with a UV laser beam **24** in irradiation step **20** represented in FIG. **1**. The beam **24** is most desirably focused by a lens **26** to a focal point within $\pm 100 \mu\text{m}$ of the front surface **102** of the glass piece **100**, most desirably within $\pm 50 \mu\text{m}$ the front surface **102**. The lens **26** has a numerical aperture desirably in the range of from 0.1 to 0.4, more desirably in the range of from 0.1 to 0.15 for

glass thickness between 0.3 mm and 0.63 mm, even more desirably in the range of from 0.12 to 0.13, so as to produce open holes extending into the glass piece **100** from the front surface **102** of the glass piece, the holes having an diameter the in range of from 5 to 15 μm , and an aspect ratio of at least 20:1. For thinner glass, in the range of from 0.1-0.3 mm, the numerical aperture is desirably from 0.25 to 0.4, more desirably from 0.25 to 0.3, and the beam is preferably focused to within $\pm 30\text{ }\mu\text{m}$ of the front surface of the glass. The laser is desirably operated at a repetition rate of about 15 kHz or below, and generally of sufficient irradiation duration to form an open hole extending just up to a back surface of the glass piece.

The irradiating step **20** of FIG. **1** is performed with a UV laser beam **24** as noted above preferably with a laser beam **24** having a wavelength in the range from 200 to 400 nm, more desirably in the range of from 300-400 nm, most preferably with an Nd:KGW (Neodymium doped Potassium-Gadolinium Tungstate) or other Nd-doped laser **22** operated at 355 nm wavelength, or within 20 nm, preferably 5 nm, thereof. The laser **22** is preferably operated at repetition rate in the range of from 5 to 50 kHz, more preferably in the range of from 10 to 20 kHz, and most preferably in the range of from 12-18 kHz.

The irradiating step **20** of FIG. **1**, in order to produce through-holes in EagleXG® glass of 0.63 mm thickness, desirably comprises irradiating the front surface **102** of the glass piece **100** with a UV laser beam **24** for a duration in the range of from 8 to 150 milliseconds per hole, more desirably in the range of from 60 to 120 milliseconds per hole, and most desirably in the range of from 80 to 100 milliseconds per hole. For 0.1 mm thick glass, about 10 milliseconds is desirable, for 0.15 mm glass 25 milliseconds, and for 0.3 mm 30 milliseconds. Thicker glass requires longer exposure (higher pulse count). As an alternative embodiment, the duration desirably may be chosen by experiment or calculation, or by a combination thereof, to be that duration which will cause the resulting hole to extend just up to a back surface **104** of the piece of glass **100**. This will allow the method to apply to various glasses having differing behavior under UV laser irradiation.

After irradiation, the resulting high aspect ratio open holes may desirably be etched in an etching step, step **40** of FIG. **1**, in an HF+HNO₃ solution. HF+HF+HNO₃ has been shown in the present work to allow for even etching across substrates with thousands of holes spaced at minimum pitch of as low as 200 μm , in contrast to some other etchant solutions. A desirable concentration is a 20% HF+10% HNO₃ solution.

As an optional step, after irradiating **20** and before etching **40**, a polishing step **60** may be applied to the front surface **102** of the glass piece **100**.

FIGS. **2** and **3** diagrammatic representation of a laser-exposure setup and an etching station, respectively, useful in the methods of the present disclosure.

The glass piece **100** desirably may placed on a motorized XYZ stage as shown in FIG. **2**, which has the accuracy and the repeatability equal or better than 1 μm . The laser beam **24** is focused with a lens **26** onto the front surface **102** of the glass **100**. The numerical aperture of the lens should ideally be more than approximately NA=0.1. Our beam **24** using a lens **26** with an aperture of NA=0.125 produced well-defined damage.

The currently preferred laser conditions are: 15 kHz repetition rate, 1.5 W mean power, and duration of 90 ms. At higher repetition rates the damage does not have well-defined boundaries as shown in FIG. **9**, which is an image of a comparative process where the repetition rate was 100 kHz. Such damage does not result in a quasi-cylindrical hole profile,

rather a conical one, at best. Powers lower than about 1.5 W do not produce enough damage while powers above 1.5 W can cause significant front-surface damage, which also leads to a funnel-type hole profile. A 90-ms train/burst of 15-kHz pulses is selected for the exposure and the burst duration is optimized for the damage extending from one side of the glass to the other, in Corning EAGLE XG® glass of 0.63 mm thickness. Longer bursts cause strong rear-surface damage, while shorter burst results in the damage-length decrease.

These laser conditions produce open or hollow micro-channels of only 7-10 μm diameter in glass, as shown in the image of FIG. **4**. Compared with laser damage that has micro-cracks, such as that produced by the comparative process shown in FIG. **9**, the micro-channel of FIG. **4** provides much better control over the etched hole profile.

The position of the focus of the beam **24** plays a significant role in damage formation. Damage like that shown in FIG. **4** can be achieved when the beam is focused within $\pm 100\text{ }\mu\text{m}$ from the front glass surface. For better consistency, this range should be reduced to $\pm 50\text{ }\mu\text{m}$. If the beam is focused closer to the back surface or behind it, the damage looks different, as shown in the image of FIG. **10**, and it becomes almost impossible to etch the hole without a waist, as shown in the image of FIG. **11**.

Under the preferred etching conditions (20% HF+10% HNO₃ by volume solution in water, 10-12 min. etching in an ultrasonic bath at approximately 35 C.^o), the resulting holes **120** are quasi-cylindrical, as seen in the image of FIG. **6**, and basically meeting the requirements stated above. Adding a surfactant to the acid, such as Capstone FS-10, for example, may help to flush the products of etching from glass. The top and the bottom views displayed in FIGS. **7** and **8**, as well as the side view image of FIG. **6**, are of holes resulting when the optional step of front side polishing **60** is employed. In the sample shown in the images, the front surface was polished by approximately 80 μm in order to the remove front-surface damage, prior to the etching step. The front-side opening of the hole can have a more irregular shape without such polishing.

FIG. **3** is a schematic representation of acid etch bench **42** useful in the etching step **40**. In FIG. **3**, the bench **42** includes an outer tub **44** with a sonic energy transmission fluid **46**, such as water, held therein. An acid tub **48** is supported with the fluid **46**, and an acid or acid blend **50** is contained therein. The irradiated and annealed glass sheet **100** is submerged in the acid or acid blend **50**. Sonic energy is applied by energy transducers **52** to the outer tub **44** and is transmitted through the intervening tubs and fluids to the glass sheet **100** during the etching process.

Etching determines the diameter of the etched hole **120** and its shape. For example, if etching is done using low acid concentrations (1% HF+1% HCl solution by volume in water) for 1 hr., the holes **120** are much smaller. The bottom diameter is 19 μm and the top diameter is 65 μm . Under these conditions the glass thickness was decreased by 10 μm from 0.63 mm to 0.62 mm. Using higher acid concentrations produced the holes of about 100 μm in diameter shown in FIGS. **6-8**. The etching parameters affecting the resulting hole dimensions include acid concentrations, etchant recipe (or selection of acids), duration of etching, and the temperature of the solution. Acid blends can include HF alone (1-30 vol %), or combined with HCl (1-50 vol %), H₂SO₄ (1-20 vol %), HNO₃ (1-40 vol %), and H₃PO₄ (1-40 vol %). The temperature of the acids preferably ranges from 25 to 60° C. Sonication or other type of agitation (such as spray-etching, for example) is desirable for solution convection within micro-holes, and for faster etching in the waist area.

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The proposed approach also enables formation of angled holes. If the laser beam is directed onto the glass sample at an angle the damage and the etched hole will be also oriented at angle to the surface. The configuration of the laser setup may be designed in such a way that it will allow for making an array, which has both holes perpendicular to the glass surface and the angled ones, such as the holes **120** shown in the schematic cross section of a glass sheet **100** in FIG. **12**.

Beam-shaping may also be used, if desired, as a means to change the hole shape, to various shapes such as shown schematically in holes **120** on glass sheet **100** of FIG. **13**. Elliptical holes have been produce by irradiating with an elliptical beam, and other shapes are possible by beam shaping through apertures and imaging, overlapping beams, and/or other techniques and combinations of techniques.

Reducing the exposure duration enables making blind holes in addition to the through-holes described above, including both hole types on the same substrate, if desired, as also shown in FIG. **12**. Blind holes will develop if, for example, the laser burst duration is reduced from 90 ms to approximately 10-20 ms. The resulting damage is similar with respect to the 7-10 μm micro-channel described above, starting at the front surface of the glass and extending to some length inside the glass, which is a function of the ratio between the shortened duration and the full duration. Etching of such a track will produce a blind hole. A combination of through and blind holes of different depth within the same hole array.

Applying an acid-resistant film/coating to the glass surfaces can improve the hole shape even further. This coating may perform several functions: (a) protect the surface from the laser-ablated debris; (b) mitigate mechanical damage to the surface of the glass surrounding the exposed area; (c) prevent glass thinning during etching thus improving the hole aspect ratio. Such coating/film may be removable or it may be left on the glass if it does not prevent further processing.

It is noted that terms like “preferably,” “commonly,” and “typically,” when utilized herein, are not utilized to limit the scope of the claimed invention or to imply that certain features are critical, essential, or even important to the structure or function of the claimed invention. Rather, these terms are merely intended to identify particular aspects of an embodiment of the present disclosure or to emphasize alternative or additional features that may or may not be utilized in a particular embodiment of the present disclosure.

Having described the subject matter of the present disclosure in detail and by reference to specific embodiments thereof, it will be apparent that modifications and variations are possible without departing from the scope of the invention defined in the appended claims. More specifically, although some aspects of the present disclosure are identified herein as preferred or particularly advantageous, it is contemplated that the present disclosure is not necessarily limited to these aspects.

It is noted that one or more of the following claims utilize the term “wherein” as a transitional phrase. For the purposes of defining the present invention, it is noted that this term is introduced in the claims as an open-ended transitional phrase that is used to introduce a recitation of a series of characteristics of the structure and should be interpreted in like manner as the more commonly used open-ended preamble term “comprising.”

What is claimed is:

1. A method of fabricating a high-density array of holes in glass, the method comprising:
providing a glass piece having a front surface;
irradiating the front surface of the glass piece with a UV laser beam, the beam being focused by a lens within

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+/-100 μm of the front surface of the glass piece, the lens having a numerical aperture in the range of from 0.1 to 0.4, so as to produce open holes extending into the glass piece from the front surface of the glass piece, the holes having an diameter the in range of from 5 to 15 μm , and an aspect ratio of at least 20:1.

2. The method according to claim **1** wherein irradiating comprises using a laser beam with a wavelength in the range of from 200-400 nm.

3. The method according to claim **1** wherein irradiating comprises using a laser beam with a wavelength in the range of from 300-400 nm.

4. The method according to claim **3** wherein the near-UV laser used in the irradiating step is operated at 355 nm wavelength, plus or minus 5 nm.

5. The method according to claim **1** wherein the numerical aperture of the lens used in the irradiating step is within the range of from 0.1 to 0.15 for glass having a thickness in the range of 0.3 to 0.63 mm.

6. The method according to claim **1** wherein the numerical aperture of the lens is within the range of from 0.25 to 0.4 for glass having a thickness in the range of 0.1-0.3 mm.

7. The method according to claim **1** wherein the laser is operated during the irradiating step at repetition rate in the range of from 5 to 50 kHz.

8. The method according to claim **7** wherein the laser is operated during the irradiating step at repetition rate in the range of from 10 to 20 kHz.

9. The method according to claim **8** wherein the laser is operated during the irradiating step at repetition rate in the range of from 12-18 kHz.

10. The method according to claim **1** wherein the step of irradiating comprises irradiating the front surface of the glass piece with a UV laser beam for a duration in the range of from 8 to 150 milliseconds per hole.

11. The method according to claim **1** wherein the step of irradiating comprises irradiating the front surface of the glass piece with a UV laser beam for a duration in the range of about 10 milliseconds per hole for glass having a thickness of about 0.1 mm.

12. The method according to claim **1** wherein the step of irradiating comprises irradiating the front surface of the glass piece with a UV laser beam for a duration of about 10 milliseconds per $\frac{1}{10}$ mm of glass thickness.

13. The method according to claim **1** wherein the piece of glass is a glass sheet, and wherein the step of irradiating comprises irradiating the front surface of the glass sheet with a UV laser beam for a duration determined by experiment or calculation to be that duration which will cause the resulting hole to extend just up to a back surface of the glass sheet.

14. The method according to claim **1** wherein the step of irradiating comprises irradiating the front surface of the piece of glass with a UV laser beam for a duration determined by experiment or calculation to be that duration which will cause the resulting hole to extend up to a location within the piece of glass.

15. The method according to claim **1** wherein the step of irradiating comprises irradiating with a laser beam having other than circular shape.

16. The method according to claim **1** wherein the step of irradiating comprises irradiating with a laser beam oriented at an angle other than 90 degrees to the front surface of the piece of glass.

17. The method according to claim **1** wherein the method further comprises etching the glass piece in acid.

18. The method according to claim **17** wherein the step of etching in acid further comprises etching in an acid blend.

19. The method according to claim **18** wherein the acid blend comprises a solution of 20% HF+10% HNO₃.

20. The method according to claim **1** further comprising, 5
after irradiating and before etching, polishing the front surface of the glass piece.

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